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Identification of Selected Silicate Minerals and their Asbestiform Varieties by Electron Optical and X-Ray Techniques

by John L. Miller, Emissions Measurement and Characterization Division, Environmental Sciences Research Laboratory*, Triangle Park, North Carolina

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16. ABSTRACT

Illustrations of electron micrographs showing morphology, electron diffraction patterns, energy dispersive X-ray spectra, and X-ray diffraction patterns of selected silicates and their asbestiform varieties are presented as an aid in their identification. A method for preparing rock quarry specimens for the quantification of choysotile asbestos is described.

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JOURNAL ARTICLE

Identification of Selected Silicate Minerals and their Asbestiform Varieties by Electron Optical and X-Ray Techniques

by John L. Miller, Emissions Measurement and Characterization Division, Environmental Sciences Research Laboratory*, Triangle Park, North Carolina

Introduction

Growing awareness in recent years of the potential danger of contracting cancer from ingesting or respiring asbestos particles has caused increased interest in the identification and quantification of asbestos-related minerals. The latest studies show that exposure to concentrated sources of asbestos even for a month can cause cancer or asbestosis 15 years later.

Where sufficient quantity of sample is available, identification of serpentine and amphibole minerals may be accomplished with X-ray diffraction and the petrographic light microscope. X-ray diffraction establishes the mineral class and the light microscope affords the morphological character. However, because of ultrasmall size, many of the airborne and waterborne serpentine and amphibole particles require electron microscopy for identification and quantification. Only one of the serpentine minerals—chrysotile—is readily identifiable by morphology, (and in some instances this is questionable); therefore, some other means is required to substantiate identity. Analytical transmission electron microscopes are equipped with selected area electron diffraction (SAED) and energy dispersive X-ray spectroscopy (EDX) capabilities for this purpose. SAED is used to establish crystalline type and EDX is used to establish elemental composition. The X-ray intensity ratios of the elements in the spectrum aid in identifying the specific mineral. To establish identity quickly and positively, the operator must have at his disposal a set of mineral specimen standards or a ready reference containing the morphology, SAED pattern, and EDX spectrum from each of the asbestos-related minerals. In the event that X-ray diffraction is used, a reference consisting of a strip chart recording of the diffraction pattern from each of the minerals is of great value [though not essential, since an American Society for Testing and Materials (ASTM) card index is generally available]. The following description of experimental procedures and results is presented as an aid in establishing the identity of an individual serpentine or amphibole mineral using electron and X-ray optical techniques.

Sample Preparation

Airborne samples are generally collected on membrane filters, using either personal samplers or high-vol samplers. Waterborne samples are collected in clean flasks and the water filtered through membrane filters in the laboratory. Samples not collected on Nuclepore filters are redeposited, after ashing and resuspension, on 0.1 μm Nuclepore membrane filters. Filters are then coated with carbon using a vacuum evaporator. Three-mm-diameter disks are punched from the carbon-coated filters, placed on electron microscope grids, and the filter material removed with a suitable solvent. If the loading on the filter is too light or too heavy, the loading is adjusted by combustion of the filter and sample in a low temperature asher followed by sample resuspension and refiltration using either an aliquot or a smaller filter area. Details of this methodology are presented elsewhere.[1]

The following preparation method is used to quantify asbestiform minerals in quarry rock samples:

Rock fragments should be from ½ to 1 in. in diameter. About ½ pt should be submitted to the laboratory for analysis. In the laboratory, the sample is placed in a 1-pt roller-type ball mill jar (size 000) and tumbled without balls for about 6 hr. Tumbling and mutual grinding of the rocks results in comminution of a layer of material at the rock surface and also produces a thoroughly mixed dust sample.

A small sample of the powder (~ 5 mg) from the ball mill jar is placed in a 50-mm-diameter agate mortar. Approximately 0.1 ml of amyl acetate is combined with the powder in the mortar and the mixture is ground with a no. 3 mullite pestle until the amyl acetate evaporates to dryness (approximately 20 min). Using a small stainless steel scalpel, the powder adhering to the pestle is scraped and thoroughly mixed with the powder remaining in the mortar. A 0.1- to 0.2-mg sample of this powder is weighed, with an electronic microbalance and placed in a 25-ml glass beaker. Fifteen ml of filtered deionized water containing 0.001% aerosol OT is added, and the beaker is covered tightly with paraffin film. The covered beaker is placed in an ultrasonic cleaner and the powder sample is dispersed in the water solution by sonication for 5 minutes.

A 47-mm-diameter filtering apparatus is assembled; this consists of a 47-mm 0.1-μm-pore-size Nuclepore filter backed up by a 5-μm-pore-size Millipore filter on the glass frit. Suction is applied and the filters recentered if necessary. The filter funnel is mounted, the vacuum is turned off, and suction is allowed to cease. The beaker is removed from the ultrasonic cleaner and the suspension of sample is poured into the funnel. The beaker is rinsed three times with water-surfactant solution and the rinsings added to the suspension in the funnel. Suction is applied and maintained until drainage is completed. The vacuum is turned off and the filter allowed to dry in still air.

After drying, the filter is immediately carbon coated [see (1), Section 2.3] and analysis for asbestos is carried out [as described in (1), Sections 2.4 ff]. The weight of powdered rock sample and the total deposit area of the filter are taken into account in calculating the fiber mass concentration from the transmission electron microscope (TEM) data.

Other types of dust or powder samples may be passed through a 140-mesh standard sieve and a 5-mg sample mulled as described above. The problem of the representativeness of dust samples from crystal sources is particularly difficult in connection with the quantification of asbestiform minerals because the latter generally occur as stratified masses. Little control can be exerted over the field collection; however, representativeness can be assured through subsequent reduction of a field sample in the laboratory by use of a spinning riffle as described by Allen.[1]

Instrumentation

A Philips Model 400 scanning transmission electron microscope is used in this study. The instrument is fitted with an energy dispersive X-ray fluorescence spectrometer. All single crystal X-ray data are obtained with a focused beam and an excitation voltage of 100 kV. Electron diffraction patterns are taken with camera lengths best suited for the pattern from the particular crystal being examined. Camera lengths are measured using gold electron diffraction rings.

Copper support grids are used throughout the study. The copper peak appears in the energy dispersive X-ray spectrum of each mineral particle and is used as a calibrating standard.

X-ray diffraction patterns are obtained using a Philips Model 3000 X-ray generator and vertical goniometer. Instrument settings are 40 kV and 35 mA at a goniometer rotation speed of 1°/min 2 θ. All samples are hand-picked by binocular microscope to insure the purity of the sample. Where small amounts of material are available, the smear mount technique is used to prepare the sample.[2]

Serpentine

Serpentine is the name given to a group of similar minerals (of which crysotile asbestos is the best known) whose composition is $Mg(Si_2O_5)(OH)_4$. There are three polymorphs of serpentine: antigorite—a platy variety; lizardite—a fine-grained and platy variety;

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and chrysotile—the fibrous variety. Serpentinite is a rock which consists mainly of serpentine; the sample of serpentinite used in this study is from Maryland. The transmission electron micrograph of this sample, Figure 1, (Figures 1 through 9 will be found on pages 4 through 11.) shows chrysotile fibers in addition to antigorite. To quote other investigators (i.e., Speil and Leineweber), "Pure serpentine is considered to be composed of nonfibrous antigorite or lizardite based on petrographic and X-ray examination. However, electron microscopy reveals the fact that all serpentine rocks contain significant amounts of chrysotile."[4]

Antigorite does not have a distinctive morphology; however, the SAED pattern is unique (see Figure 1) and affords a quick means of identifying this polymorph. The energy dispersive X-ray spectrum as well as the X-ray diffraction pattern seen in this figure are common to all three polymorphs.

Chrysotile is the fibrous form of serpentine. It occurs most frequently as cross-fiber veins ranging in size from microscopic to greater than six in in length. Chrysotile is also found in dolomite and dolomitic limestone where contact with basic igneous rock intrusions occurs. It is the principal asbestiform mineral used industrially.

Chrysotile has a unique morphology and SAED pattern (see Figure 2), which make it one of the easiest to identify by electron optical methods. There are instances, however, in which the crystal is partially masked by some coating or when the crystal is too small to yield a satisfactory SAED pattern, making identification difficult. In such cases, identification depends upon the morphology and the energy dispersive X-ray spectrum. The ratio of the magnesium peak to the silicon peak (0.8) in the EDX spectrum, though varying slightly from one crystal to another, is sufficient to establish identity.[5]

Depending on the source, the chemical composition of chysotile in bulk form is as follows [after Huggins[6]]:

Chrysotile (Range %)	
SiO_2	38.8-41.5
Fe_2O_3	0.04-1.6
FeO	.3-2.0
Al_2O_3	0.04-4.7
MgO	38.2-42.6
CaO	0.35-2.0
Na_2O	0.04-0.1
K_2O	0.02-0.2
H_2O+	11.4-12.9
H_2O-	0.6-0.9

Amphiboles

The literature contains excellent descriptions of the amphiboles.[4,7,8,9] These rock-forming minerals include crocidolite (which is the fibrous form of riebeckite); the fibrous forms of tremolite, actinolite, hornblende, and grunerite-cummingtonite; "amosite," (the fibrous form of grunerite, whose name derives from the acronym for Asbestos Mines of South Africa); and anthophyllite.

The chemical composition of the asbestiform amphiboles is as follows [after Speil and Leineweber[4]]:

Asbestiform Amphibole (Range %)					
	Crocidolite	Amosite	Anthophyllite	Actinolite	Tremolite
SiO_2	49-53	49-53	56-58	51-56	55-60
MgO	0-3	1-7	28-34	15-20	21-26
FeO	13-20	34-44	3-12	5-15	0-4
Fe_2O_3	17-20	-	-	0-3	0-0.5
Al_2O_3	0-0.2	-	0.5-1.5	1.5-3	0-2.5
CaO	0.3-2.7	-	z -	10-12	11-13
K_2O	0-0.4	0-0.4	-	0-0.5	0-0.6
Na_2O	4.0-8.5	-	-	0.5-1.5	0-1.5
H_2O	2.5-4.5	2.5-4.5	1.0-6.0	1.5-2.5	0.5-2.5

The chemical formula for crocidolite is $Na_2Fe_3^{2}Fe_2^{3}(Si_8O_{22})(OH)_2$. Its color is grayish blue (hence the name "blue asbestos"). The fibers are flexible, but not as much as those of chrysotile. Crocidolite has a higher tensile strength than chrysotile. Good hand specimens of the mineral have a silky appearance.

Crocidolite is found in iron-rich sedimentary rocks in cross-fiber veins, and also in sodium-rich metamorphic and igneous rocks. South Africa is the principal source of the industrial grade material.

After grinding in a ball mill, crocidolite exhibits acicular morphology (see Figure 3). Some crystals have a fibrous appearance. High magnification shows cleavage fractures of a singular nature and no bundles except those resulting from agglomeration. Selected area electron diffraction patterns are easily obtained from even the smallest crystals. The energy dispersive X-ray spectrum confirms the chemical formula given above, though the iron valency states are unobtainable by this method. The X-ray diffraction pattern is typical of this amphibole mineral.

Tremolite [$Ca_2Mg_5Si_8O_{22}(OH)_2$] is usually gray to white in color and consists of coarse, silky fibers. It occurs most commonly as long, slender needles that radiate in all directions into the adjacent rock body. Tremolite is found in a variety of rock forms, especially in dunite and peridotite. It is also commonly associated with metamorphosed marine limestones ($CaCO_3$) and dolomites [$CaMg(CO_3)_2$] that are mixed with sand and magnesium silicate minerals or dirty marbles. Tremolite is one of the most common amphiboles.

The sample (shown in Figure 4) is from Murphy, North Carolina, and is the non-asbestiform variety. This tremolite is massive, radiating prismatic crystals. For this study, single pieces are removed and ground. After crushing in a highspeed ball mill, tremolite exhibits a bladed-to-acicular morphology with an aspect ratio of from 3:1 to 10:1 (see the transmission electron micrograph in Figure 4). The selected area electron diffraction pattern reflects the (110) cleavage. The energy dispersive X-ray pattern from a single particle shows only a trace of iron. A micrograph and SAED pattern from the asbestiform variety are presented in this figure for comparison.[9]

Actinolite [$Ca_2(Mg, Fe)_5Si_8O_{22}(OH)_2$] is a high-iron member of the tremolite-actinolite series. However, the pure-iron-end member is not found; usually, the maximum iron content is of the order of 20%. Actinolite is commonly green or greenish gray, and the fibers are quite brittle. Occurrence is similar to tremolite, but with more iron-rich sediments in the vicinity.

After crushing in a ball mill, non-asbestiform actinolite exhibits a prismatic morphology (see the transmission electron micrograph in Figure 5). This variety has less tendency to fracture into long fibers or acicular crystals.

This sample of actinolite shows the presence of aluminum, which would place it compositionally near hornblende (note the EDX pattern).

Hornblende, the major constituent of amphibolite, is chemically very complex; the formula is $(Ca,Na^x)_2$ $(Mg,Fe,Al)_5$ $(Al,Si)_8O_{22}$ $(OH,F)_2$. The color of hornblende may range from dark green to black. The rare fibrous variety has a silky luster and occurs in very thin cross-fiber veins. The morphology of the non-asbestiform variety is bladed to acicular (see Figure 6) and the energy dispersive X-ray spectrum is similar to that of actinolite. The only satisfactory means of distinguishing this mineral from actinolite is X-ray diffraction analysis.

Grunerite ("amosite") is found (like crocidolite) in iron-rich sediments), but (unlike crocidolite) with little or no sodium. Its chemical formula is $(Mg,Fe)_7Si_8O_{22}(OH)_2$. Grunerite is mined for use in the asbestos industries in South Africa, but is a contaminant in iron mining operations in the Lake Superior area of the United States. It occurs as cross-fiber veins and is usually brown. The morphology of ground "amosite" is acicular to fibrous (see Figure 7). The individual crystal is lathe-shaped. Identification by selected area electron diffraction should be done using a goniometer stage and photographing the SAED pattern at several tilt angles.[10] The EDX spectrum and X-ray diffraction pattern are typical of this asbestiform mineral.

Anthophyllite $(Mg,Fe)_7(Si_8O_{22})(OH)_2$ occurs in short-fibrous, cross-fiber veins in schists; the fibers are slightly flexible. The color, depending on weathering, varies from grey-green to brown. Anthophyllite is fairly common in altered mafic igneous rocks in the Blue Ridge Mountain chain. It is the only orthorhombic fibrous amphibole. The morphology of crushed anthophyllite is bladed to acicular (see Figure 8). Its energy dispersive X-ray spectrum closely resembles that of chrysotile, except for a difference in the magnesium-to-silicon ratio.

Conclusions

The identification and quantification of asbestiform minerals is at present performed most satisfactorily using electron optical methods; this is especially true where airborne or waterborne samples containing chrysotile are concerned. Chrysotile fibrils are too small to be resolved by the light microscope, but the mineral is easily identified by its tubular morphology and unique electron diffraction pattern. When the diffraction pattern is too faint to observe or record, one may rely on the energy dispersive X-ray spectrum.

For quantification of chrysotile in serpentinite rock, careful consideration must be given to sample preparation, especially the grinding phase. Chrysotile is generally present in the rock in micro veins that tend to disintegrate into small chunks. These chunks must be given sufficient grinding time to divide into free fibers.

A well-tested methodology for the analysis of chrysotile in serpentinite rock and for the analysis of amphibole minerals in other types of rock has been outlined. Identification of these two classes of minerals (serpentine and amphibole) can be aided by use of the illustrations of morphologies, selected area electron diffraction patterns, energy dispersive X-ray spectra, and X-ray diffraction patterns. Depending on circumstances, two or more of these types of observation are generally required.

Acknowledgments

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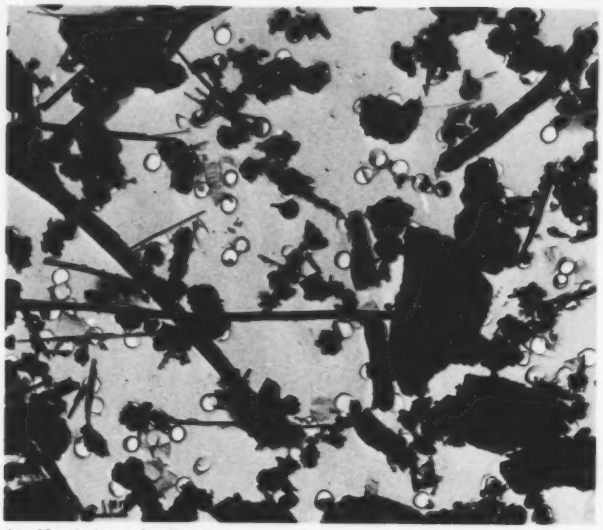
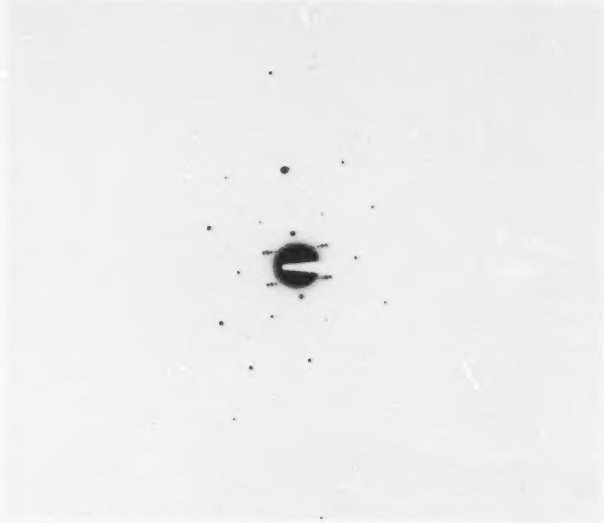
Editor's note: Figures 1 through 9 will be found on the following 8 pages.

Serpentine "Antigorite"

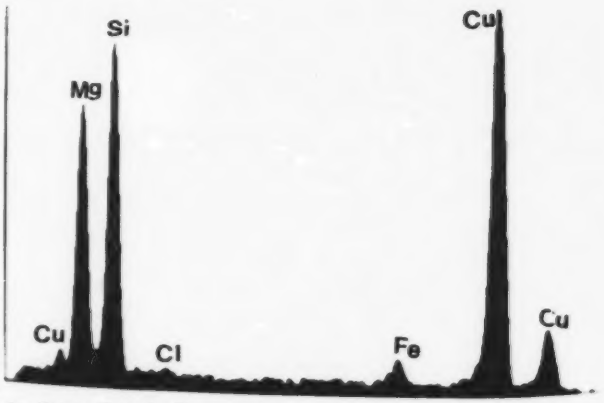


1a. Morphology of antigorite.

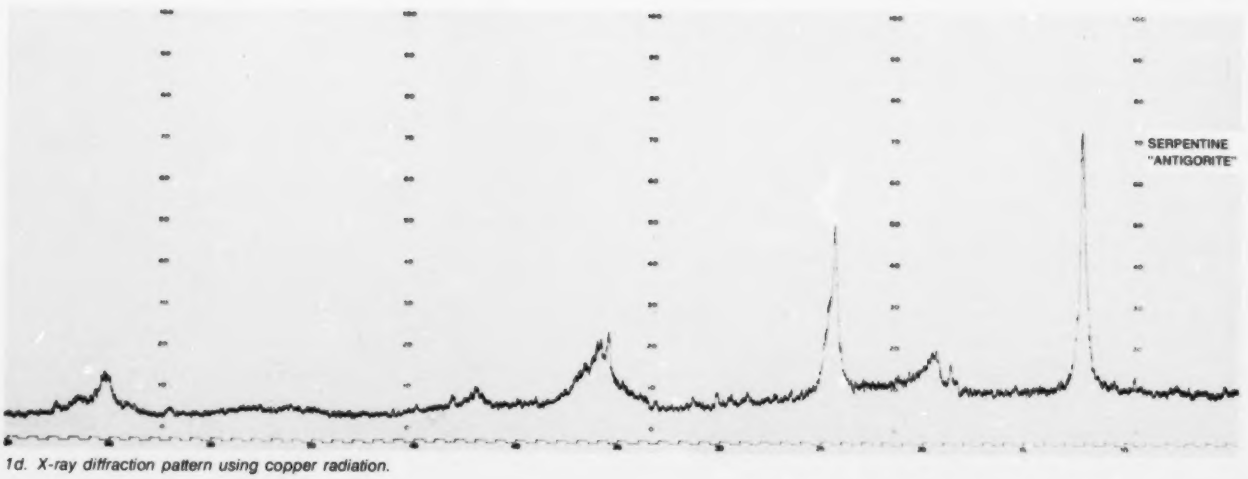


1b. Electron diffraction pattern.

Figure 1. Serpentine is the name given to a group of similar minerals with three polymorphs: antigorite, a platy variety; lizardite, a fine-grained and platy variety; and chrysotile, the fibrous variety. The micrograph (Figure 1a) shows chrysotile fibers in addition to antigorite. Antigorite does not have a distinctive morphology; however the SAED pattern is unique and affords a quick means of identifying this polymorph. The energy dispersive X-ray spectrum as well as the X-ray diffraction pattern (Figures 1b and 1c) are common to all three polymorphs.

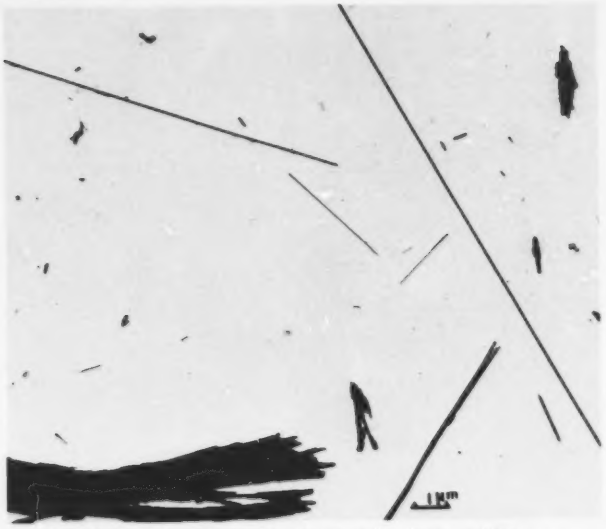


1c. Energy dispersive X-ray spectra.

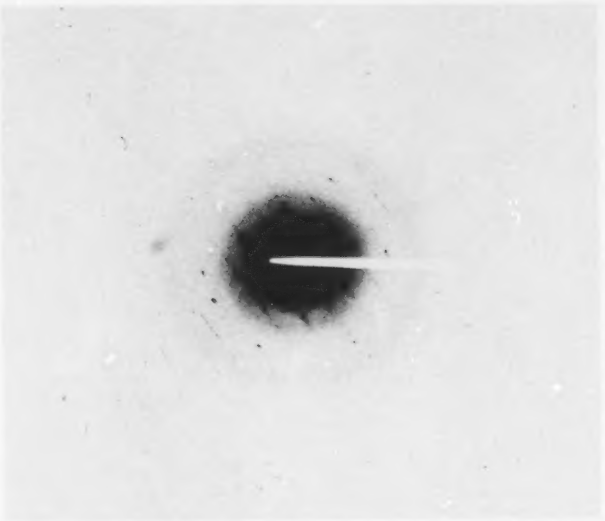


1d. X-ray diffraction pattern using copper radiation.

Chrysotile

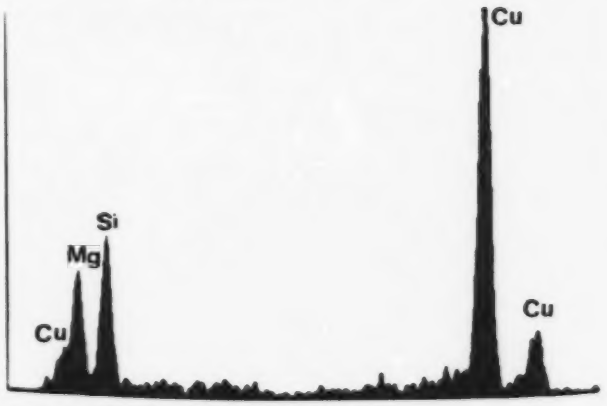


2a. Morphology of chrysotile, showing fiber bundles, fibers and fibrils.

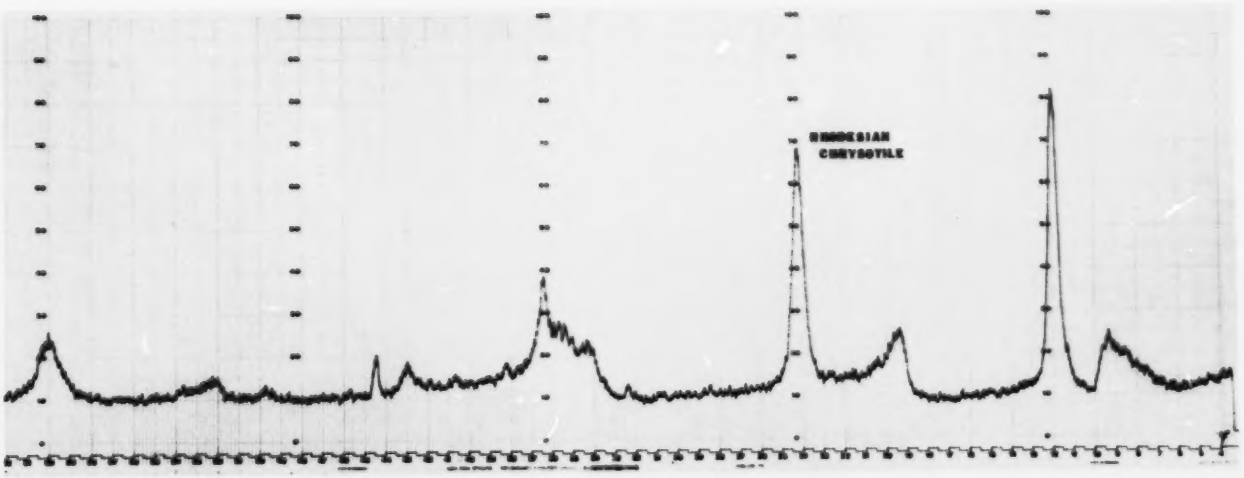


2b. Electron diffraction pattern from fibril.

Figure 2. Chrysotile is the fibrous form of serpentine and is the principal asbestiform mineral used industrially. Chrysotile has a unique morphology (Figure 2a) and SAED pattern (Figure 2b) which make it one of the easiest to identify by electron optical methods. There are instances, however, in which the crystal is partially masked by some coating or when the crystal is too small to yield a satisfactory SAED pattern. In such cases, identification depends upon the morphology and energy dispersive X-ray spectrum. The ratio of the magnesium peak to the silicon peak in the EDX spectrum, though varying slightly from one crystal to another, is sufficient to establish identity.



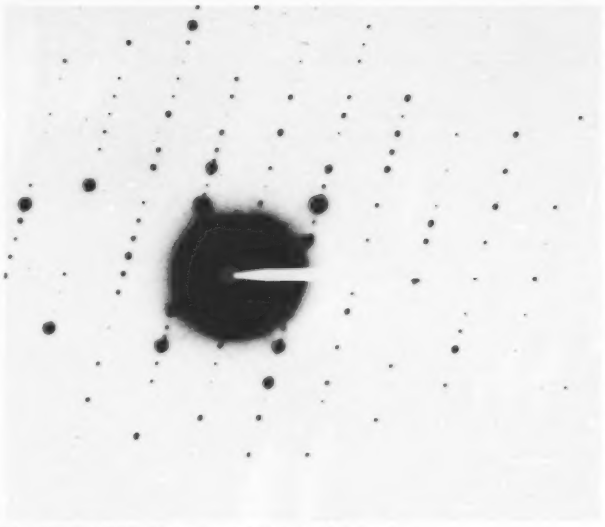
2c. Energy dispersive X-ray spectra from fibril.



2d. X-ray diffraction pattern using copper radiation.

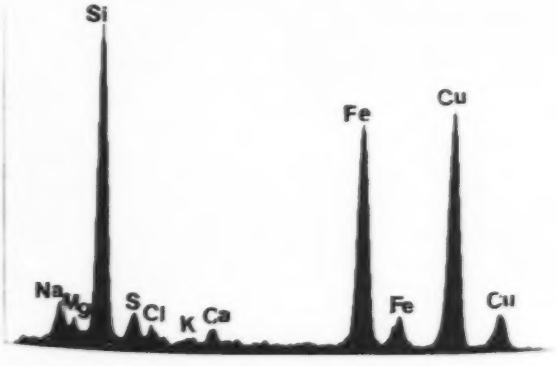
Crocidolite

3a. Morphology of crushed crocidolite.

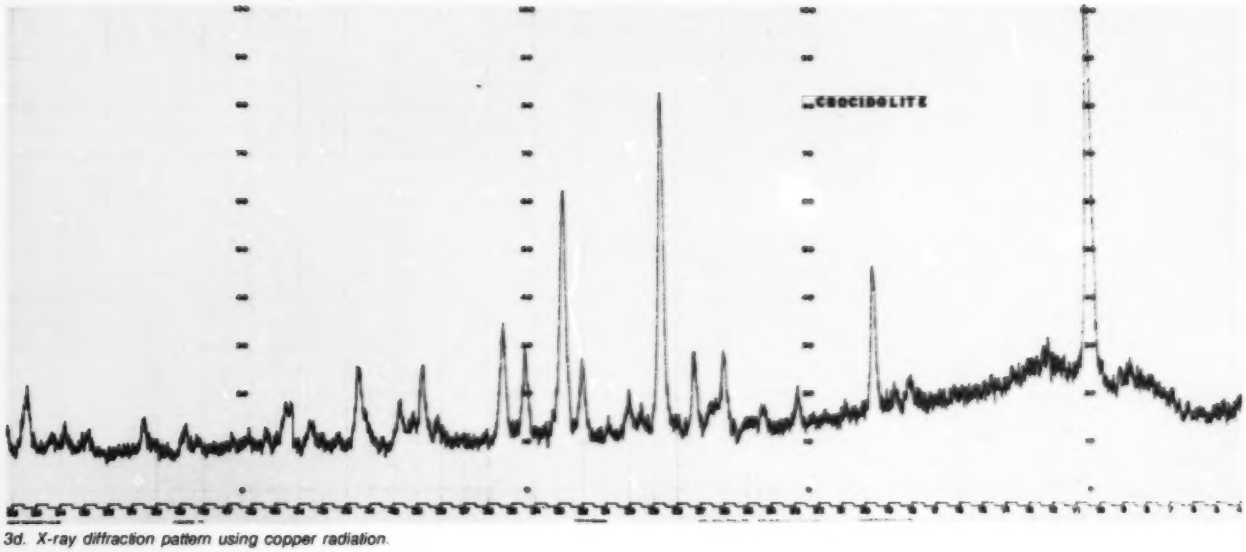


3b. Electron diffraction pattern of fiber in micrograph.

Figure 3. Crocidolite, which comes principally from South Africa, is an industrial grade mineral. After grinding in a ball mill, crocidolite exhibits acicular morphology. Some crystals have a fibrous appearance. High magnification shows cleavage fractures of a singular nature and no bundles except those resulting from agglomeration. SAED patterns are easily obtained even from the smallest crystals. The EDX spectra confirms the chemical formula, though the iron valency states are unobtainable by this method; the X-ray diffraction patterns is typical of the amphibole mineral.

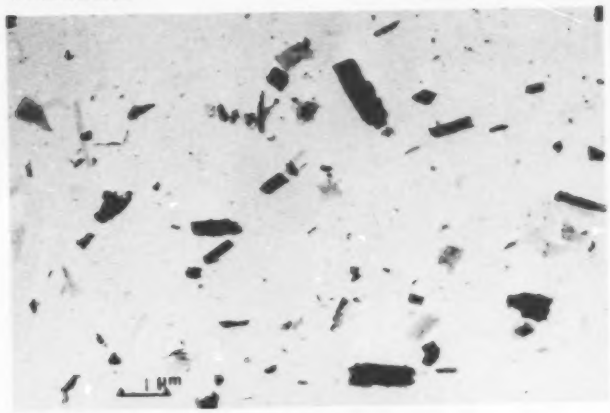


3c. Energy dispersive X-ray spectra.

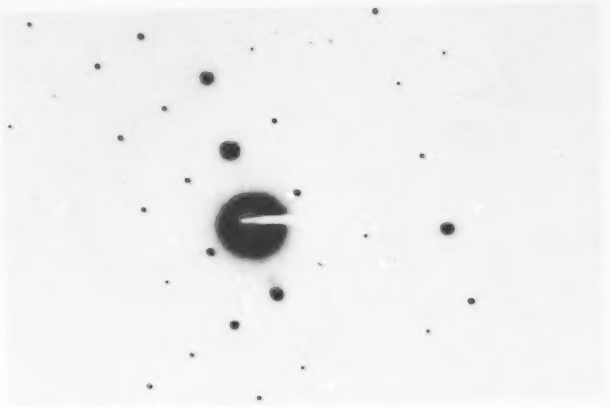


3d. X-ray diffraction pattern using copper radiation.

Tremolite

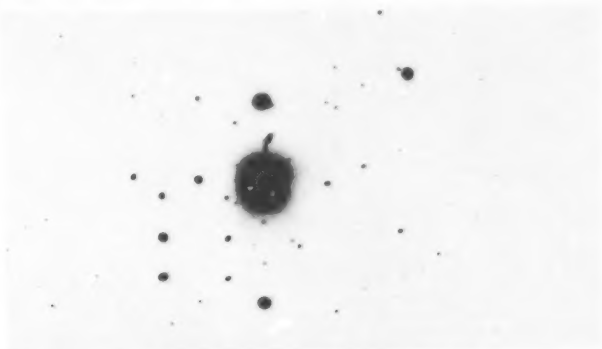


4a. Morphology of crushed non-asbestos tremolite.



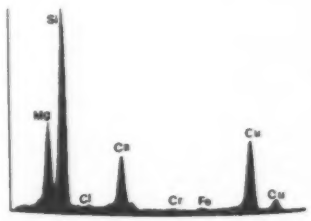
4b. Electron diffraction pattern from cleavage fragment.

4c. Morphology of fibrous variety of tremolite.

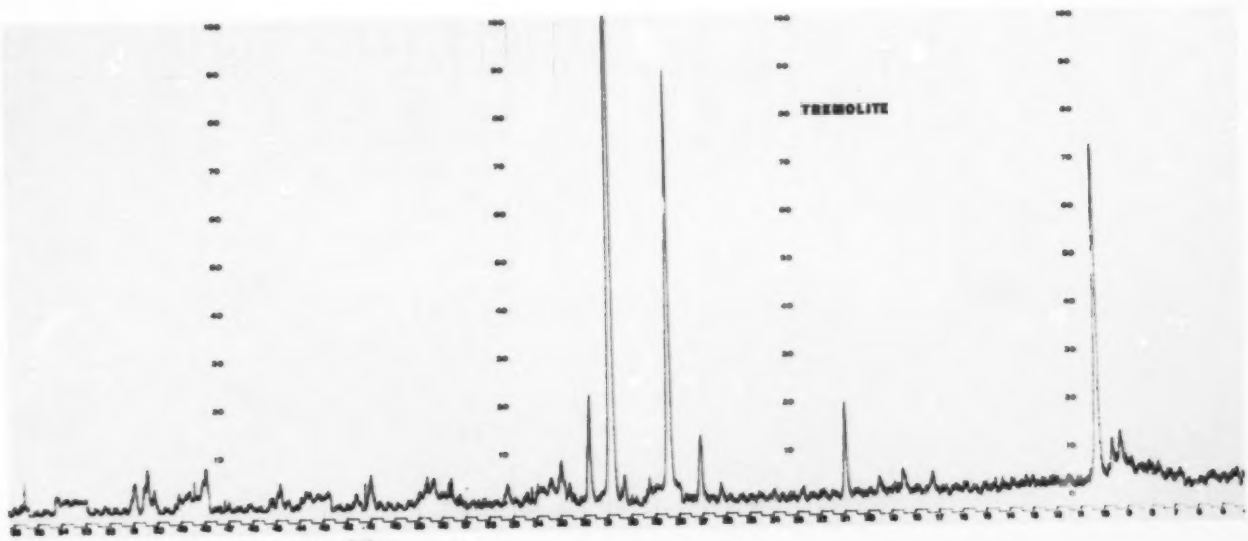


4d. Electron diffraction pattern from fiber of fibrous variety.

Figure 4. Tremolite is the most common of the amphiboles and occurs most commonly as long slender needles that radiate in all directions into the adjacent rock body. After crushing in a high speed ball mill, temolite exhibits a bladed-to-acicular morphology with an aspect ratio of from 3:1 to 10:1 (Figure 4a). The SAED pattern shows the (110) cleavage. Figures 4c and 4d show the asbestiform variety.

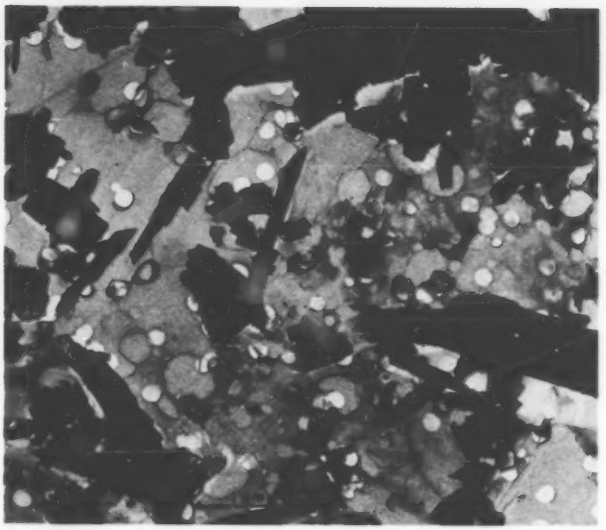


4e. Energy dispersive X-ray spectra.

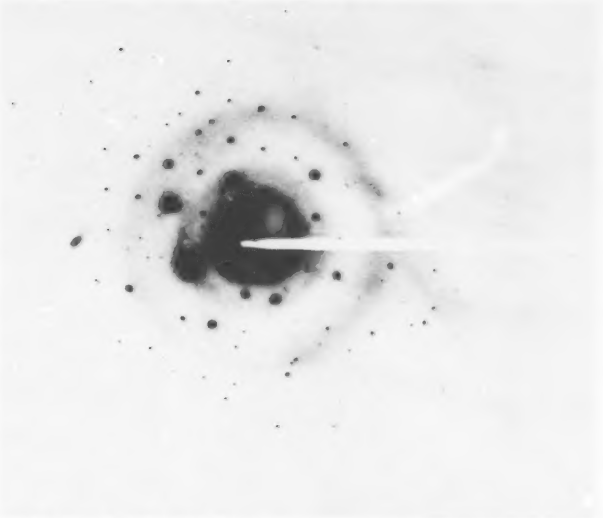


4f. X-ray diffraction pattern using copper radiation.

Actinolite

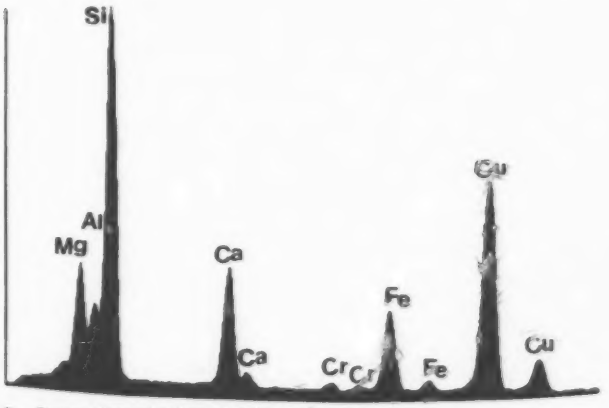


5a. Morphology of crushed actinolite.

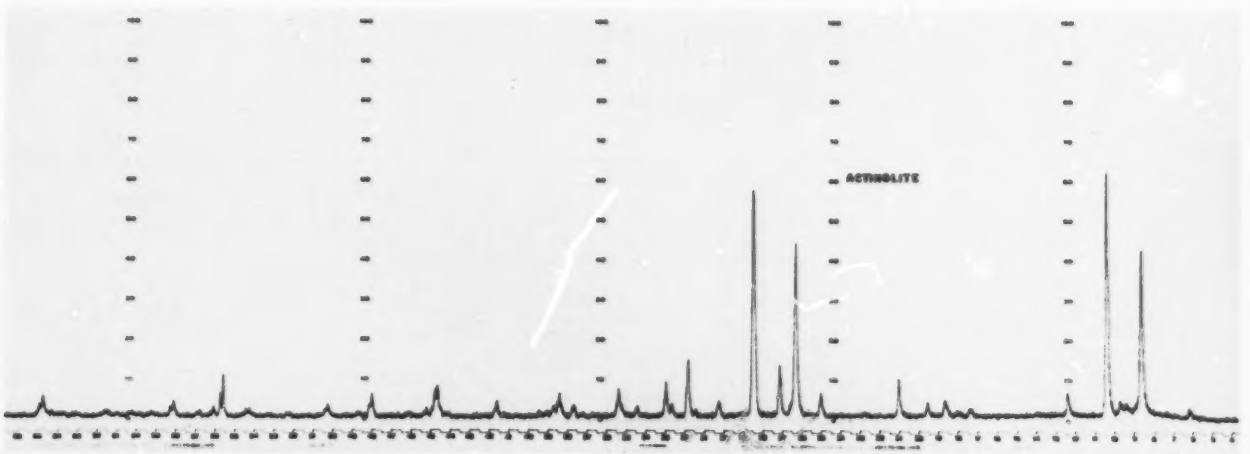


5b. Electron diffraction pattern.

Figure 5. Actinolite is a high-iron member of the tremolite-actinolite series, with the maximum iron content being in the order of 20%. After crushing in a ball mill non-asbestiform actinolite exhibits a prismatic morphology (Figure 5a).

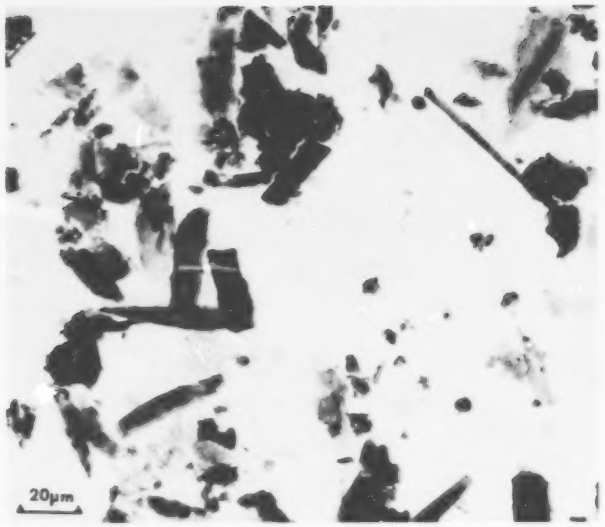


5c. Energy dispersive X-ray spectra.

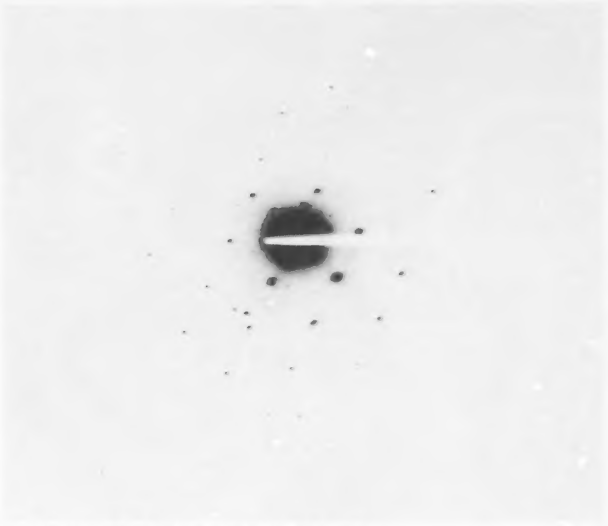


5d. X-ray diffraction pattern using copper radiation.

Hornblende

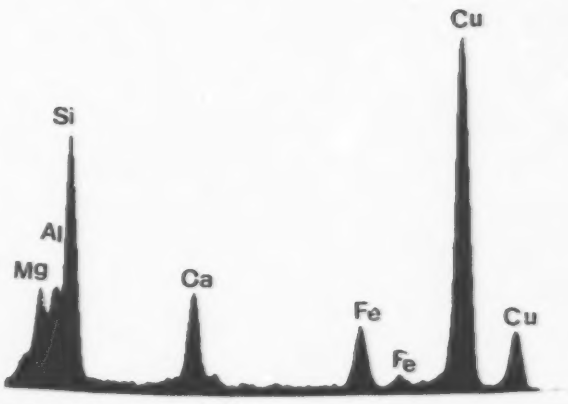


6a. Morphology of crushed hornblende.

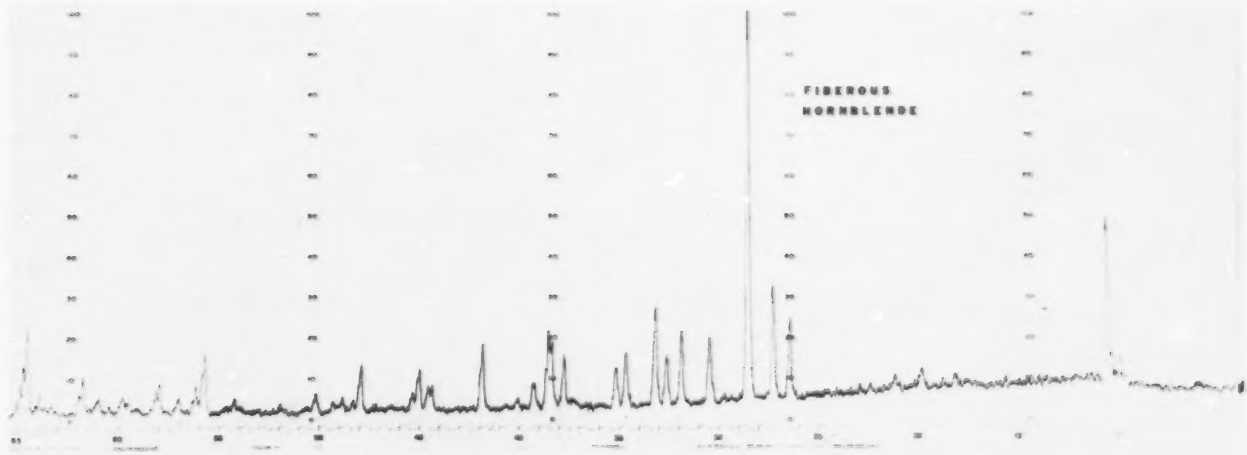


6b. Electron diffraction pattern from single crystal.

Figure 6. Hornblende is the major constituent of amphibolite. The morphology of the non-asbestiform variety is bladed to acicular (Figure 6a) and the EDX pattern is similar to that of actinolite. The only satisfactory means of distinguishing this mineral from actinolite is through X-ray diffraction analysis.

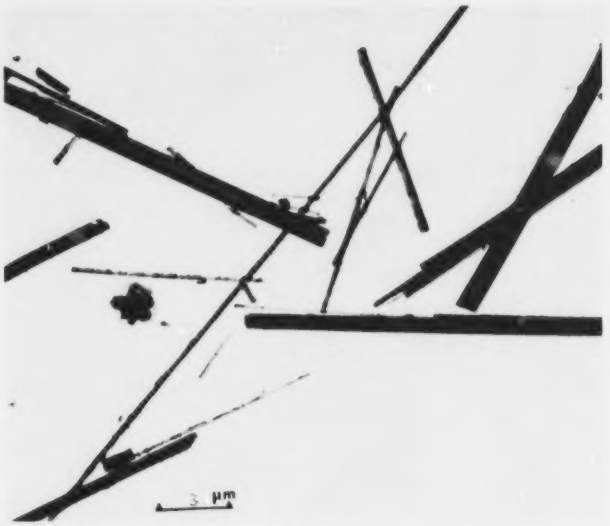


6c. Energy dispersive X-ray spectra.

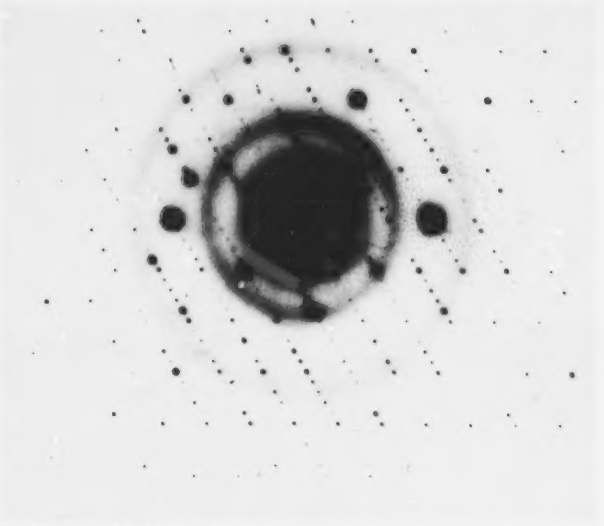


6d. X-ray diffraction pattern using copper radiation.

Grunerite "Amosite"

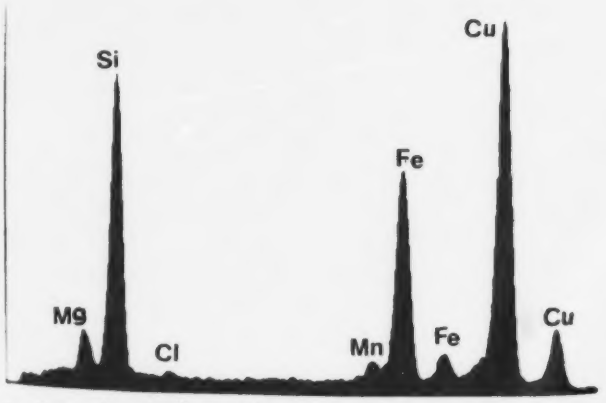


7a. Morphology of crushed "amosite".

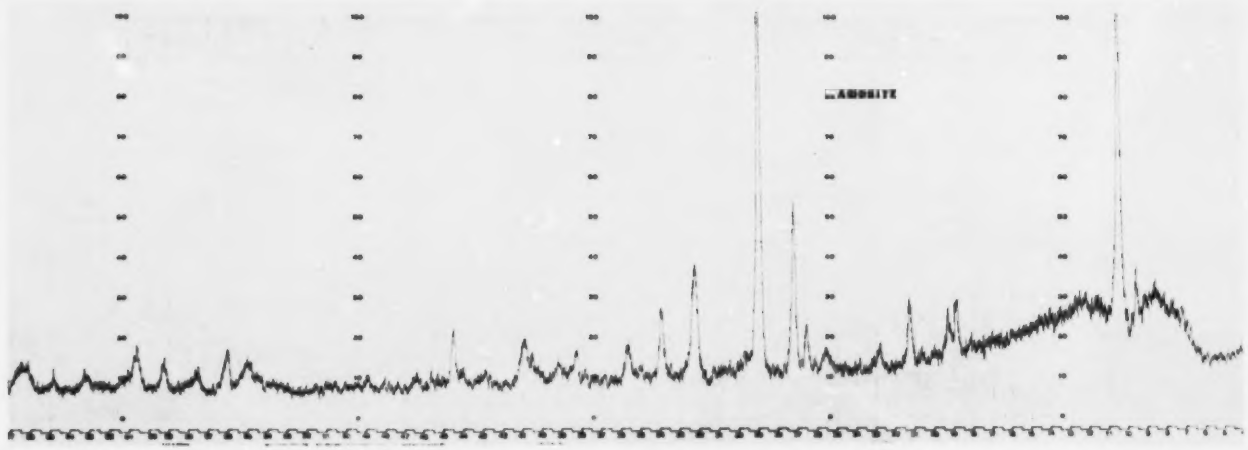


7b. Electron diffraction pattern.

Figure 7. Grunerite ("amosite") like crocidolite is found in iron-rich sediments, but unlike that mineral, contains little or no sodium. Although mined for use in the asbestos industries in South Africa it also appears as a contaminant from iron mining operations in the Lake Superior area of the U.S. It occurs as cross fiber veins and is usually brown. The morphology of ground "amosite" is acicular to fibrous (Figure 7a). The individual crystal is lath-shaped. Identification by SAED should be done using a goniometer stage and photographing the SAED pattern at several tilt angles. The EDX spectra and X-ray diffraction pattern shown are typical.

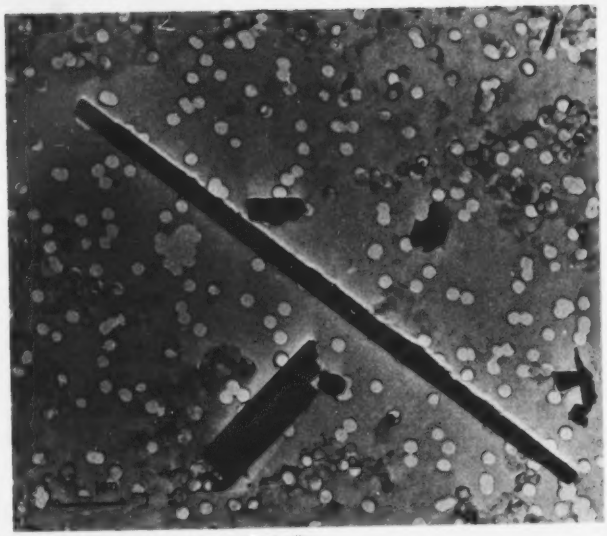


7c. Energy dispersive X-ray spectra.

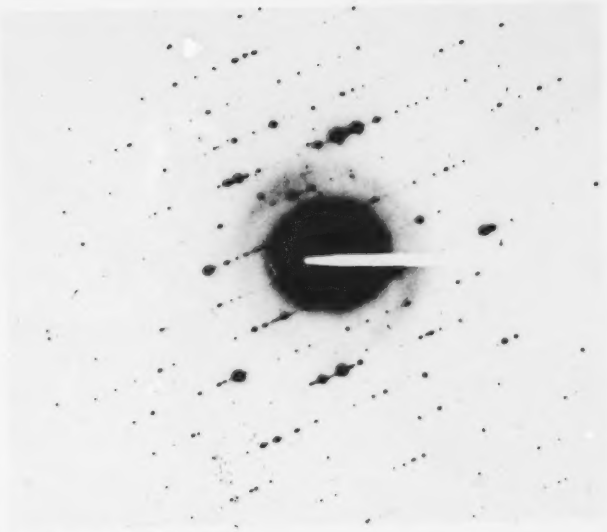


7d. X-ray diffraction pattern using copper radiation.

Anthophyllite

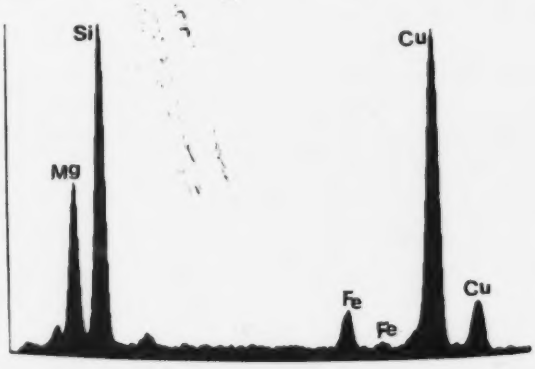


8a. Morphology of crushed anthophyllite.

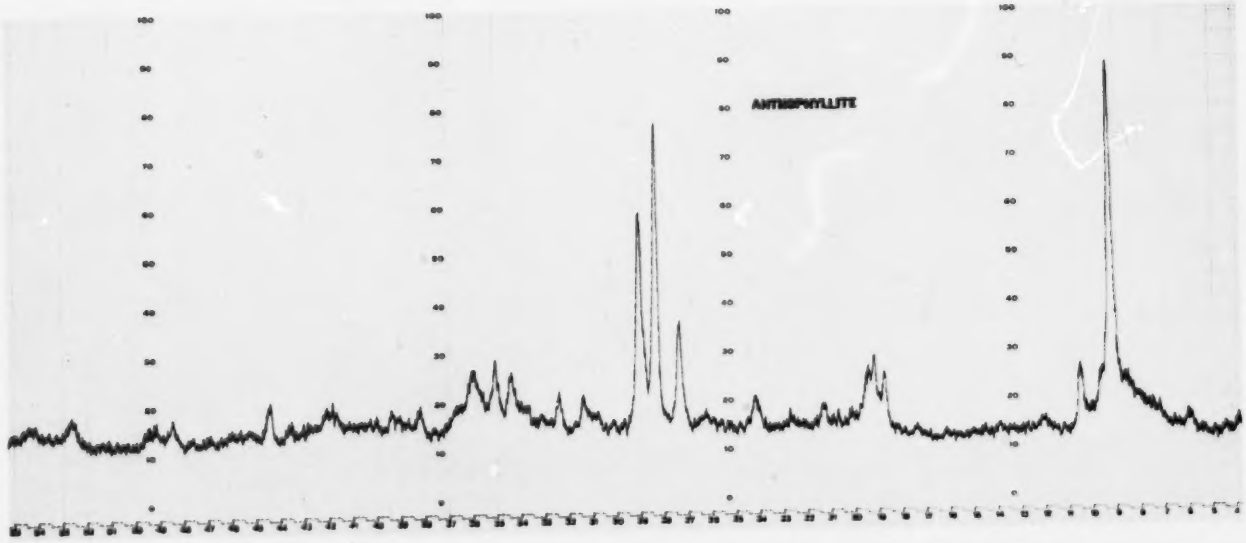


8b. Electron diffraction pattern.

Figure 8. Anthophyllite occurs in short-fibrous, cross-fiber veins in schists and is the only orthorhombic fibrous amphibole. The morphology of crushed anthophyllite is bladed to acicular (Figure 8). The EDX spectra closely resembles that of chrysotile, except for a difference in the magnesium-to-silicon ratio.



8c. Energy dispersive X-ray spectra.



8d. X-ray diffraction pattern using copper radiation.